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| 14. ABSTRACT<br>A comprehensive and systematic study has been made on $\text{Sm}(\text{Co}_{0.91}\text{Fe}_x\text{Cu}_y\text{Zr}_x)_2$ magnets to completely understand the effects of composition and processing on their magnetic properties with the aim to increase their operating temperature. The homogenized $\text{Sm}(\text{Co}, \text{Fe}, \text{Cu}, \text{Zr})_2$ magnets have a featureless microstructure. A cellular/lamellar (2:17R cells surrounded by 1:5 cell boundaries superimposed on 2:17H lamella) microstructure develops after 2-3 hours of aging at 800-850°C, but the coercivity increases only after a subsequent slow cooling to 400°C. During the cooling, diffusion takes place and Cu is concentrated in the 1:5 cell boundaries and Fe in the 2:17R cells. Our studies have led to compositions with operating temperatures above 400°C and with coercivities as high as 10 kOe at 500°C <sup>1</sup> . The high temperature performance of these magnets is very sensitive to the Cu content, which is believed to control both the coercivity and its temperature dependence. Furthermore, we developed a more economical heat treatment processing for higher Cu content magnets. |             |  |                            |   |   |
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F49620-96-1-0434

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# **UD MURI PROGRAM**

**ON**

## **MAGNETS FOR HIGH TEMPERATURE APPLICATIONS**

**Final Report**

**Date: February 20, 2002**

# MAGNETS FOR HIGH TEMPERATURE APPLICATIONS

## OBJECTIVES

The aim of this work was to develop high temperature magnets to be used in an electromagnetic system for the "More Electric Plane" applications. The desired operating temperature  $T_{op}$  was 450°C for permanent magnets and 650°C for soft magnets.

## HIGHLIGHTS OF ACCOMPLISHMENTS

- Completely understood the effects of the complex magnet composition and processing on the magnetic properties of  $\text{Sm}(\text{Co}, \text{Fe}, \text{Cu}, \text{Zr})_z$  magnets. We can now consistently design magnets with tailored properties.
- Obtained the first cast  $\text{Sm}(\text{Co}, \text{Fe}, \text{Cu}, \text{Zr})_z$  high temperature magnets with coercivity at 500°C greater than 10 kOe. These magnets are now made commercially.
- Discovered a simpler and more economical processing route for magnets with higher Cu and Zr content.
- First to observe and explain the abnormal temperature dependence of coercivity  $H_c(T)$ , with the coercivity increasing with temperature in magnets with a low Cu content.
- Modeled the coercivity behavior in terms of repulsive and attractive domain wall pinning.
- Developed new 2:17 magnets based on the  $\text{Y}(\text{Co}, \text{Fe}, \text{Cu}, \text{Zr})_z$  and  $\text{Pr}(\text{Co}, \text{Fe}, \text{Zr})_z$  compounds, despite the fact that  $\text{Y}_2\text{Co}_{17}$  and  $\text{Pr}_2\text{Co}_{17}$  have planar anisotropy and therefore were not expected to be suitable for PM development.
- Systematically studied the magnetic and mechanical properties of different bulk Co-Fe alloys. The effects of grain size, ordering, precipitates and defects on the magnetic properties are now well understood.
- Developed W fiber reinforced Co-Fe composites which have the mechanical properties for the Air Force applications

## BRIEF SUMMARY OF RESEARCH RESULTS

### Part A : Permanent Magnets

#### Effects of Composition on Magnetic Properties

The  $\text{Sm}(\text{Co}_{bal}\text{Fe}_v\text{Cu}_y\text{Zr}_x)_z$  magnets represent a complicated system with four compositional variables ( $x, y, v, z$ ). The fully developed magnets consist of a cellular microstructure consisting of 2:17R cells surrounded by 1:5 cell boundaries which is superposed on a 2:17H lamellar structure (Fig. 1). This microstructure is developed after a complex heat treatment consisting of a homogenization at 1100-1200°C, followed by an aging at 800-850°C and then a slow cooling to 400°C (Fig. 2). During the slow cooling to 400°C, the microstructure does not change but the microchemistry does with the Cu diffusing to the 1:5 cell boundaries leading to the observed high  $H_c$  (Fig 3). The decrease of ratio  $z$  leads to the formation of more 1:5 cell boundaries and thus to the reduction of cell size (Fig. 4) For a fixed Cu content this means that there is more Cu in the 1:5 cell boundaries of high  $z$  magnets because the 1:5 cell boundaries are fewer and therefore  $H_c$  is larger. At higher  $Z$ , the cells start breaking and  $H_c$  decreases. Figure 5 shows the

temperature dependence of coercivity  $H_c(T)$  of  $\text{Sm}(\text{Co}_{\text{bal}}\text{Fe}_{0.1}\text{Cu}_{0.08}\text{Zr}_{0.04})_z$  magnets with different ratio  $z$ . The magnets with lower ratio  $z$  have a lower RT coercivity and better  $H_c(T)$ .

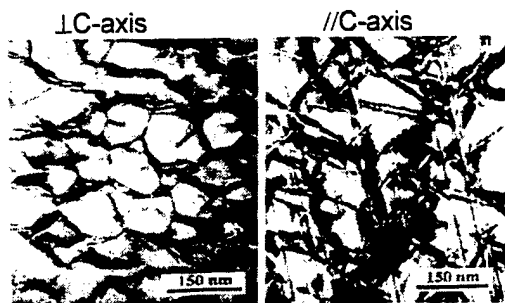


Fig. 1 typical cellular/lamellar microstructure

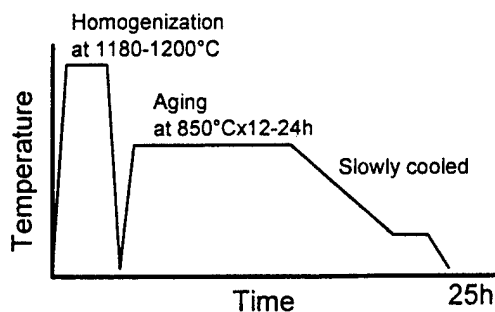


Fig. 2 Schematic of heat treatment

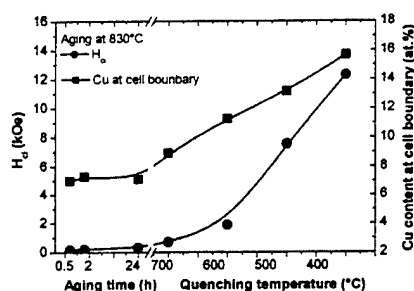


Fig.3 Development of Cu content in the cell boundaries and  $H_c$  with

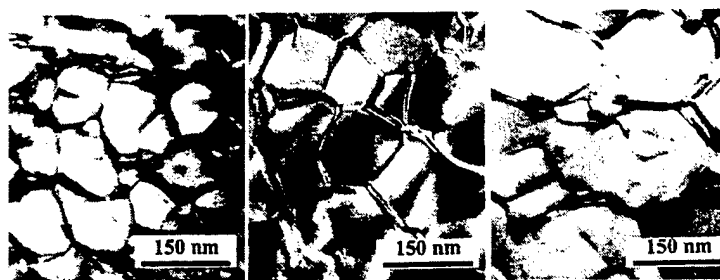


Fig. 4 Cellular microstructure of  $\text{Sm}(\text{Co}_{\text{bal}}\text{Fe}_{0.224}\text{Cu}_{0.08}\text{Zr}_{0.033})_z$  magnets with (a)  $z=7.0$ ,  $H_{ci}=15$  kOe, (b)  $z=8.5$ ,  $H_{ci}=35$  kOe and  $z=9.1$ ,  $H_{ci}=8$  kOe.

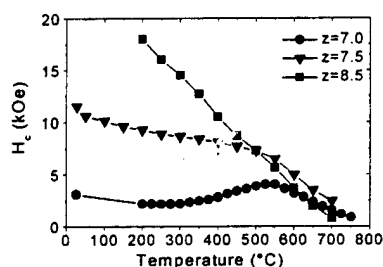


Fig. 5 Temperature dependence of coercivity  $H_c(T)$  of  $\text{Sm}(\text{Co}_{\text{bal}}\text{Fe}_{0.1}\text{Cu}_{0.08}\text{Zr}_{0.04})_z$  magnets with different ratio  $z$ .

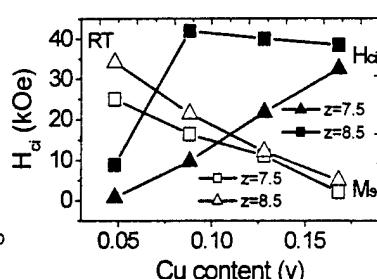


Fig. 6  $M_s$  and  $H_{ci}$  of  $\text{Sm}(\text{Co}_{\text{bal}}\text{Fe}_{0.1}\text{Cu}_y\text{Zr}_{0.04})_z$  magnets ( $y=0.048-0.168$ ,  $z=7.5$  and  $8.5$ ) on the Cu content

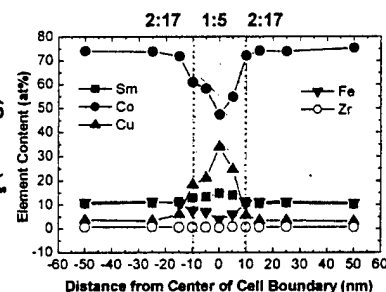


Fig. 7 Element profile in the 1:5 cell boundary

Cu content has the most important effect on the microstructure and magnetic properties of the magnets. Figure 6 shows the dependence of saturation magnetization  $M_s$  and coercivity  $H_{ci}$  of  $\text{Sm}(\text{Co}_{\text{bal}}\text{Fe}_{0.1}\text{Cu}_y\text{Zr}_{0.04})_z$  magnets ( $y=0.048-0.168$ ,  $z=7.5$  and  $8.5$ ) on the Cu content.  $M_s$  decreases while  $H_{ci}$  increases with increasing Cu content. Our nanoprobe chemical analysis results (Fig. 7) show that the Cu concentration at the cell boundaries increases with increasing the Cu content of the magnets. Because the magnet with low ratio  $z$  has a smaller cell size, the proportion of the cell boundaries is larger and more Cu is needed to obtain the saturated value of coercivity (Fig. 6).

The addition of Zr leads to the formation of lamella phase which stabilizes a uniform cellular microstructure with the right microchemistry for high coercivity. Figure 8 shows  $H_c$  as a function of Zr content. With increasing Zr content, the coercivity dramatically increases and reaches a value of around 40 kOe for  $x$  in the range from 0.02 to 0.06, and the cell size decreases, first slightly and then quickly, from 120 to 35 nm (Fig. 9).

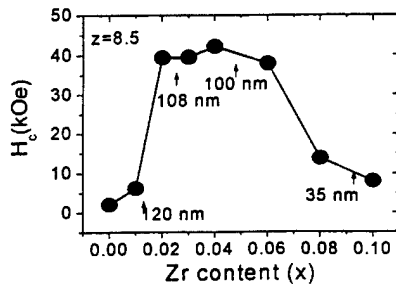


Fig. 8  $H_c$  as a function of Zr content for  $\text{Sm}(\text{Co}_{0.91}\text{Fe}_{0.09}\text{Cu}_{0.088}\text{Zr}_x)_{8.5}$  magnets.

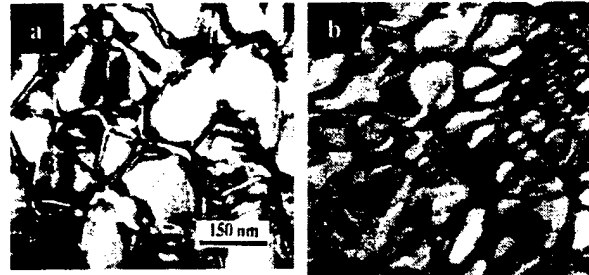


Fig.9 TEM cellular microstructures for the  $\text{Sm}(\text{Co}_{0.91}\text{Fe}_{0.09}\text{Cu}_{0.088}\text{Zr}_x)_{8.5}$  magnets with (a)  $x=0.04$  and (b)  $x=0.08$

The addition of a certain amount of Fe is necessary to develop a uniform cellular microstructure with a larger cell size and thus a high coercivity. Figure 10 shows the effect of Fe content on the magnetic properties of magnets with different compositions. TEM studies (Fig. 11) show that the microstructure exhibits a transformation from a smaller but non-uniform cell size to a more uniform one. For a fixed Cu, this leads to a larger Cu content in the 1:5 cell boundaries and therefore, to a higher coercivity.

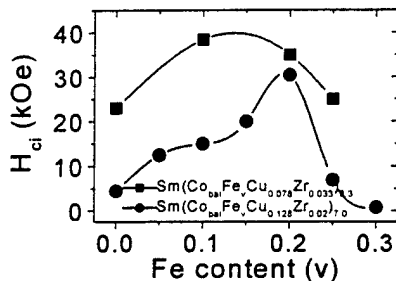


Fig. 10  $H_c$  as a function of Fe content

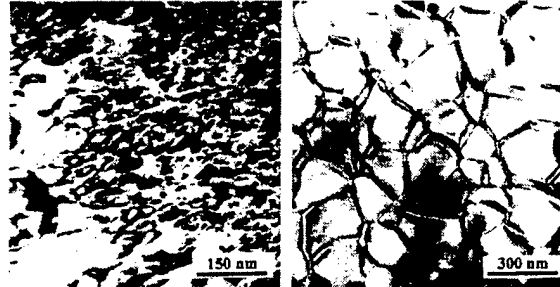


Fig. 11 TEM micrographs of  $\text{Sm}(\text{Co}_{0.91}\text{Fe}_v\text{Cu}_{0.128}\text{Zr}_{0.02})_{7.0}$  magnets (a)  $v=0$ , (b)  $v=0.02$

### The First Cast $\text{Sm}(\text{Co}, \text{Fe}, \text{Cu}, \text{Zr})_z$ High Temperature Magnets with High $H_c$

Using the knowledge derived from the above studies, we are able to finely tune the microstructure and microchemistry of the Sm-Co magnets through adjustments in the composition and processing parameters, and design magnets for various applications. Figure 12 show the best high temperature magnets. Magnets A exhibits a  $H_c$  of 10 kOe at 500C and a very good temperature stability.

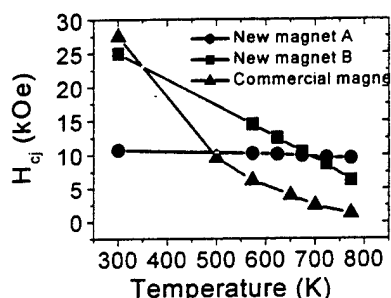


Fig. 12 Temperature dependence of  $H_c$  of typical magnets

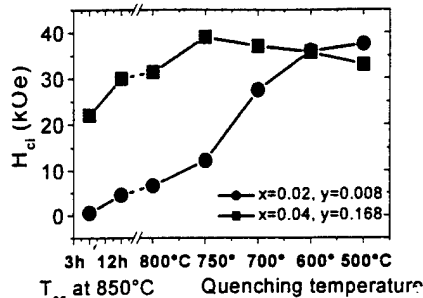


Fig. 13. Development of  $H_c$  with aging of  $\text{Sm}(\text{Co}_{0.98}\text{Fe}_{0.1}\text{Cu}_{0.168}\text{Zr}_{0.04})_{3.5}$  magnets

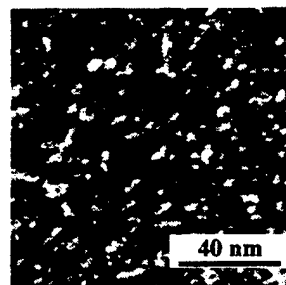


Fig. 14 TEM image of solutionized  $\text{Sm}(\text{Co}_{0.98}\text{Fe}_{0.1}\text{Cu}_{0.168}\text{Zr}_{0.04})_{3.5}$  magnet

### New Aging Cycle for Fabricating Sm-Co Magnets

For certain  $\text{Sm}(\text{Co}_{0.98}\text{Fe}_{0.1}\text{Cu}_{0.168}\text{Zr}_{0.04})_{8.5}$  alloys with higher Cu content, a room temperature  $H_c$  of 22 kOe and  $H_c$  of 6.3 kOe at 500°C are obtained only after aging for 3h at 850°C followed by quenching (Fig. 13). The microstructure of these magnets shows that in the solutionized state, 1:5 phase precipitates are observed in a 2:17H matrix instead of the featureless 2:17H, which facilitates the rapid development of cellular microstructure (see Fig. 14) without the need of aging.

### Abnormal Temperature Dependence of Coercivity and Coercivity Mechanism

Abnormal  $H_c(T)$  always exists in magnets with the cellular (or cellular-like) microstructure regardless of the presence of Cu. Figure 15 shows the  $H_c(T)$  of  $\text{Sm}(\text{Co}_{0.98}\text{Fe}_{0.1}\text{Cu}_y\text{Zr}_{0.04})_z$  magnets. For a given  $z$ , there exists a critical Cu amount. The abnormal  $H_c(T)$  is observed when the Cu content is lower than the critical value. This critical Cu amount increases with decreasing ratio  $z$ . The reason for this behavior is related to the changes of 1:5 intrinsic magnetic properties with Cu and temperature.

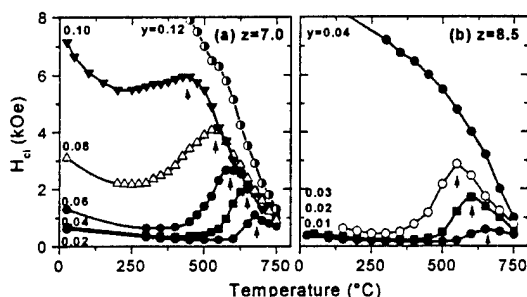


Fig. 15  $H_c(T)$  of magnets with different  $z$  and Cu content

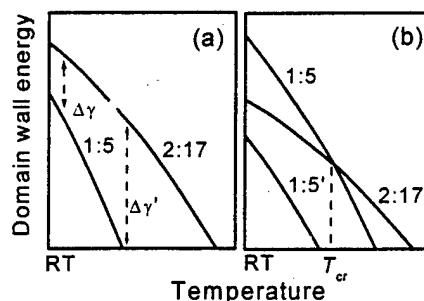


Fig. 16 A schematic of  $\gamma$  as a function of temperature of 2:17 and 1:5 phases

The variation of Cu content in 1:5 cell boundaries results in the change of the anisotropy and Curie temperature of the 1:5 boundary phase. Figure 16 shows a schematic of the temperature dependence of domain wall energy of the 2:17 and 1:5 phases with different Cu content. At  $T_{cr}$ ,  $\gamma_{1:5} = \gamma_{2:17}$ . Cu substitution in the 1:5 phase leads to the gradual reduction of a critical temperature  $T_{cr}$ . Domain wall pinning explains the abnormal behavior as a temperature-induced transition at  $T_{cr}$  from a low  $H_c$  repulsive DW pinning ( $\gamma_{1:5} \gg \gamma_{2:17}$  in the case of lower Cu content,  $\gamma$  is domain wall energy density) to a high  $H_c$  attractive pinning ( $\gamma_{2:17} \gg \gamma_{1:5}$ , in the case of higher Cu content).

### New 2:17 Magnets Based on the $Y(Co,Fe,Cu,Zr)_2$ and $Pr(Co,Fe,Zr)_2$ Compounds

New 2:17 magnets based on the  $Y(Co,Fe,Cu,Zr)_2$  and  $Pr(Co,Fe,Zr)_2$  compounds have been developed. As shown in Fig. 17, the microstructure of these magnets shows the similar cellular morphology like that in the Sm-Co 2:17 magnets. The typical hysteresis loops are shown in Fig. 18, 19, respectively. The Y-Co based magnets can be produced as sintered magnets, or as anisotropic powders. The magnets are expected to be less expensive, easier to produce, and have enhanced corrosion resistance. Pr-Zr-Co is one of a few (if not the only) anisotropic nanocomposite exchange-coupled magnet. The orthogonal easy directions of  $Pr_2Co_{17}$  and  $PrCo_5$  cause unique orientation phenomena with an increased  $H_c$  in the aligned Pr-Zr-Co magnet.

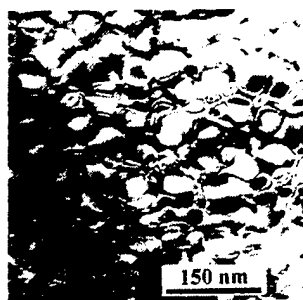


Fig. 17 TEM image of  $Y(Co,Fe,Cu,Zr)_2$  compounds

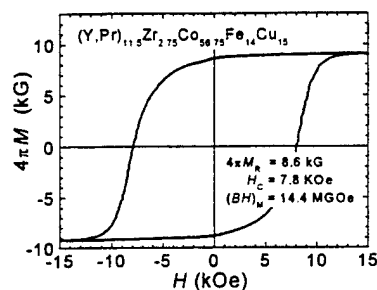


Fig. 18 Hysteresis loop of  $Y(Co,Fe,Cu,Zr)_2$  compounds

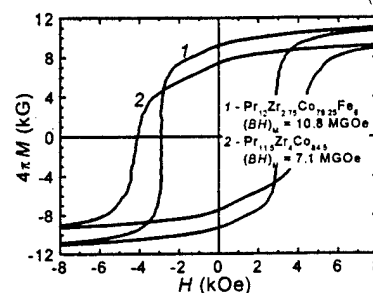


Fig. 19 Hysteresis loop of  $Pr(Co,Fe,Zr)_2$  compounds

## Part B: Soft Magnets

Our efforts in soft magnets were focused on the following three projects; (i) Bulk Co-Fe alloys, (ii) Fiber reinforced Co-Fe composites and (iii) nanocrystalline Finemet-type ribbons. A summary of the objectives and current status in the first two projects is shown in Table 1.

Table 1 Program objectives and current status

|                                     | AF requirement<br>Rotor                                 | Hiperco 50HS   | Fiber-Metal<br>Compsites                |
|-------------------------------------|---|--|---|
| Operating<br>Temperature            | 600°C<br>5000 hr  | 600°C  | 600°C                                   |
| Induction<br>requirement            | 2 T<br>500°C  | 2.06/1.98 T<br>600°C/aged                            | >2T<br>600°C                            |
| Yield<br>Strength                   | 700 MPa   | 350 MPa  | > 700 MPa                               |
| Creep Rate<br>Tempearture<br>Stress | 5.6×10 <sup>-10</sup> /sec<br>550°C<br>600 MPa (87 ksi) | 7×10 <sup>-6</sup> /sec<br>550°C<br>600 MPa (87 ksi) | 0 (>200hr)<br>550°C<br>600 MPa (87 ksi) |
| Resistivity                         | 40-60 μΩ-cm   |  |   |
| Core Loss<br>Frequency              | 480 W/kg<br>5000 Hz                                     | 48 W/kg<br>400 Hz                                    | N/A                                     |

### Bulk Co-Fe Alloys

We have systematically investigated the high temperature magnetic and mechanical properties of FeCo alloys. The microstructural factors such as grain size, precipitates, defects, and structural order parameter significantly change both the magnetic and mechanical properties of FeCo-based alloys, especially in samples with small additions of V and Nb (Fig. 1-2). We have experimentally found that grain boundaries, precipitates, and disordered phases act as pinning sites for the magnetic domain wall movement,

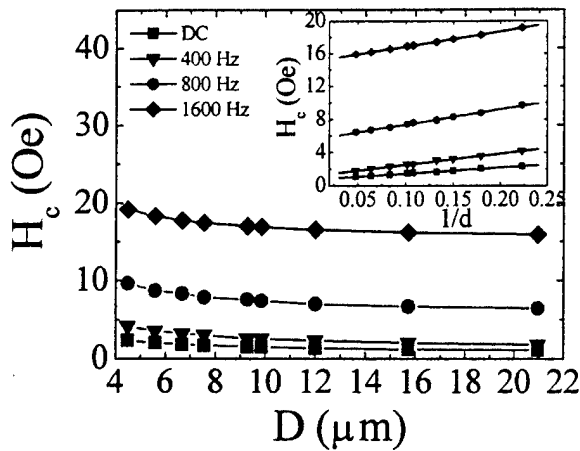


Fig. 1 The grain size dependence of magnetic properties

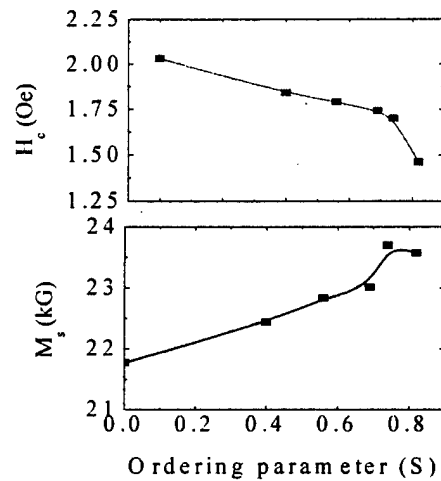


Fig. 2 Dependence of magnetic properties on ordering parameter

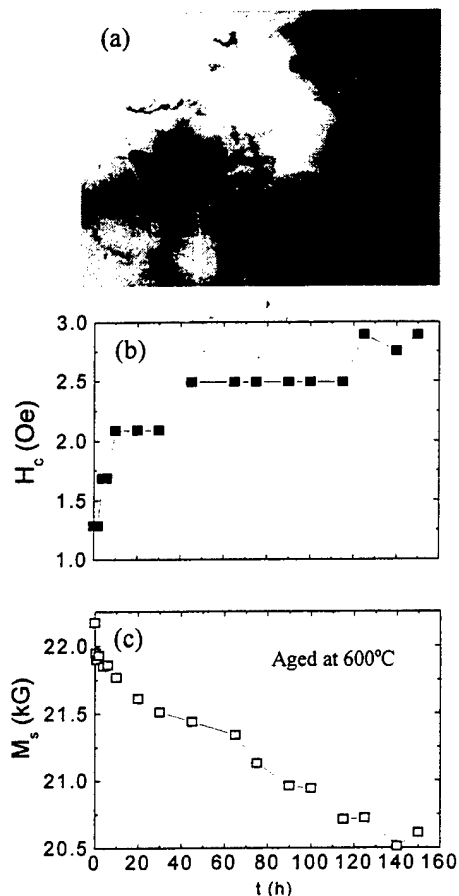


Fig. 3. Lorenz microscopy graph of magnetic domains pinned by second phases (a), and the effect of aging time on the magnetic properties of Hiperc050 alloys (b and c).

are not very good consisting of poor yield strength, ductility and creep behavior at high temperature (Table 1)

### Co-Fe composites

To significantly improve the mechanical properties without considerably sacrificing the magnetic properties, we have designed and fabricated fiber and ceramic particle reinforced soft magnetic composites (Fig. 4). Excellent mechanical properties including high yield strength and low creep rate have been observed in the fabricated fiber composites (Fig. 5). These magnetic composite materials show promising potential in applications and open a new avenue for the exploration of the interesting physical phenomena.

### Nanocrystalline Finemet-type

This study was performed to determine the suitability of Cobalt substituted Finemet for potential high temperature applications. Finemet was chosen as the starting point for

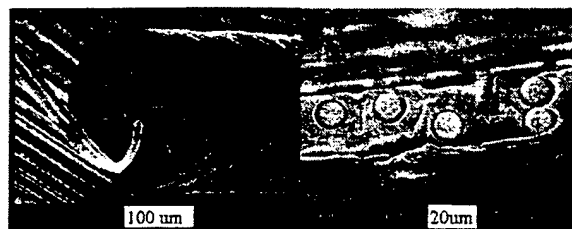


Fig. 4 SEM observations on fiber reinforced soft magnetic composites.

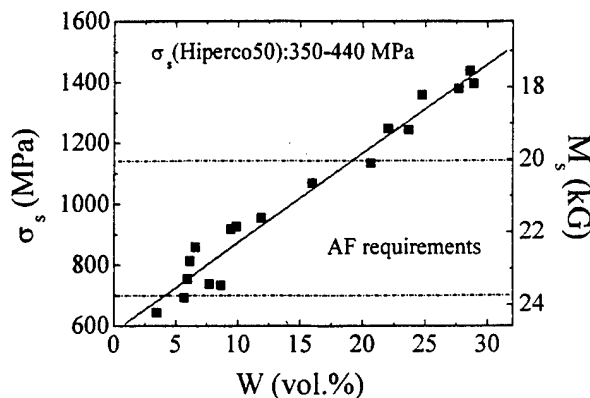


Fig.5 Mechanical properties of fiber reinforced composites

which increase the coercivity  $H_c$ , whereas the precipitates and order parameters alter the saturation magnetization  $M_s$  (Fig. 3). The precipitation of second phase creates most adverse effects on magnetic properties. The mechanical properties of bulk commercial FeCo-base alloys

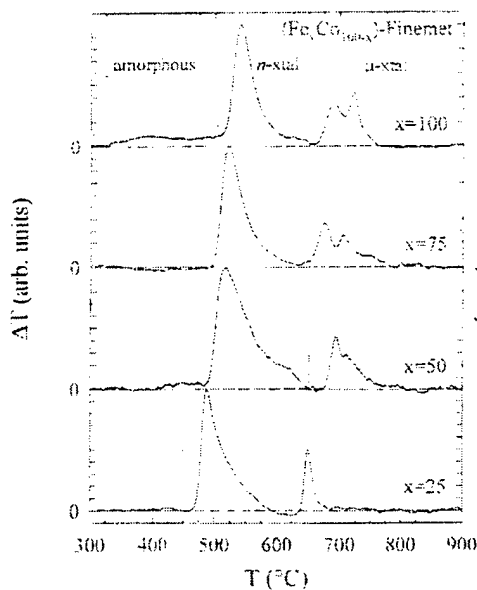


Fig. 6 DTA results on  $(\text{Fe}_x\text{Co}_{100-x})$ -Finemet alloys.

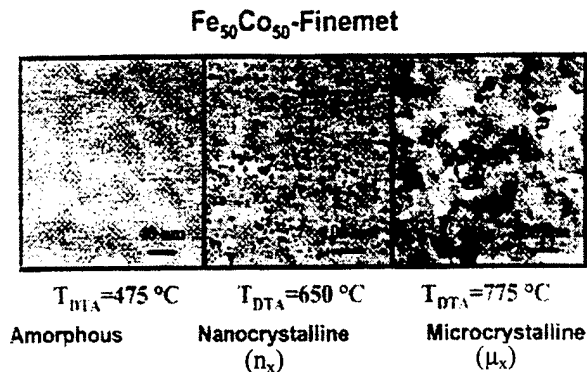


Fig. 7 TEM observation of structural evolution in  $\text{Fe}_{50}\text{Co}_{50}$ -Finemet alloys

this study because of its magnetic softness (room temperature coercivity around 0.01 Oe) and kinetic stability with time for annealing temperatures in the range of 517 to 607 °C. However, it has undesirable temperature dependencies of magnetization and coercivity because of dilution effects and grain decoupling at elevated temperatures near the amorphous Curie temperature around 330 °C. Cobalt substitutions were chosen to reduce the dilution effect and increase the amorphous Curie temperature.

Thermal and structural studies indicate that there are in general three exothermic thermal events (Fig. 6). The first one involves the precipitation of nanocrystalline BCC grains (Fig. 7). This is followed by the crystallization of the amorphous matrix. Finally, the nanocrystalline BCC grains undergo a secondary crystallization and become microcrystalline (Fig. 7). The first thermal event therefore defines the temperature range for crystallization into an amorphous nanocrystalline composite. Activation energies decrease monotonically from about 4.7 eV/atom for  $x = 100$  to about 3.5 eV/atom for  $x = 25$ . The onset temperatures for crystallization also decrease monotonically from about 525 °C for  $x = 100$  to about 475 °C for  $x = 25$ . These results suggest that the thermal stability of these alloys decreases with increasing cobalt substitution. The temperature dependence of magnetization for each alloy indicates that the grains and amorphous matrix are ferromagnetic. (Fig. 8) The Curie temperature of grains asymptotically increases for each alloy with increasing Co content. While the Curie temperature of amorphous phase decreases for each alloy. Based on the lattice parameters and grain Curie temperatures, the grains for  $x = 100$

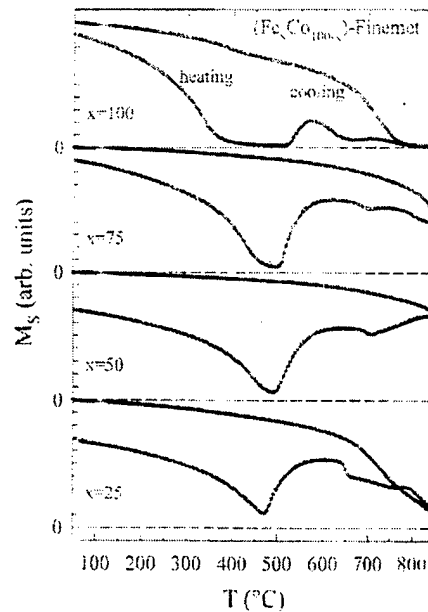


Fig. 8 The temperature dependence of magnetization of Co-substituted Finemet alloys

are initially an Fe(SiBNb) solid solution and evolve to Fe<sub>80</sub>Si<sub>20</sub> at. %. Further, it is believed the cobalt substituted grains are initially an Fe(CoSiBNb) solid solution and evolve to Fe<sub>57</sub> to 90Co<sub>0</sub> to 19Si<sub>10</sub> to 24 at. %. Assuming that the asymptotic composition contains 20 at. % silicon, there would be approximately 10 at.% cobalt and 70 at.% iron.

The temperature dependence of coercivity for each alloy shows an increase near the amorphous Curie temperature as expected (Fig. 9). This increase washes out with crystallization because the intergranular distance decreases and therefore the grains couple independent of the amorphous matrix. The alloys don't exhibit superparamagnetic behavior or superferromagnetic behavior as calculated by Herzer. Although certain aspects of Herzer's model for the temperature dependence of coercivity are in qualitative agreement with experimental results, there are several aspects that are not and need to be reevaluated.

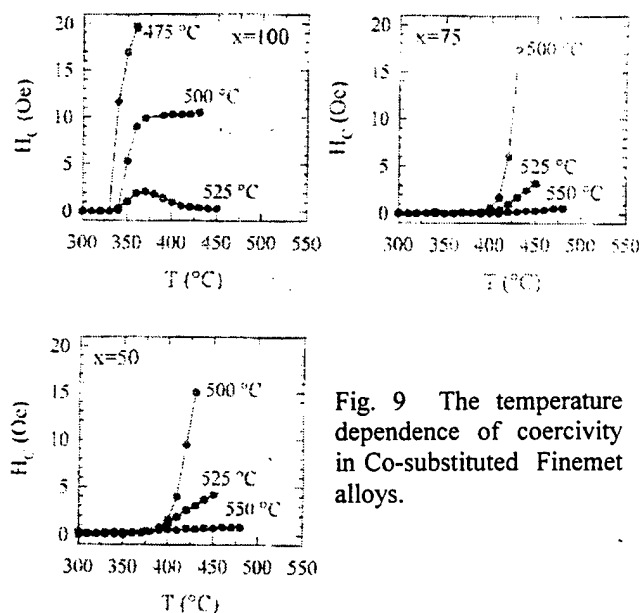


Fig. 9 The temperature dependence of coercivity in Co-substituted Finemet alloys.

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